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APPLICATIONS OF SYNCHROTRON RADIATION TO MATERIALS SCIENCE:  
DIFFRACTION IMAGING (TOPOGRAPHY) AND MICRORADIOGRAPHY

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ABSTRACT

Synchrotron radiation sources are now available throughout the world. In this paper, the use of hard x-ray radiation from these sources for materials science is described with emphasis on diffraction imaging for material characterization. With the availability of synchrotron radiation, real-time in situ measurements of dynamic microstructural phenomena have been started. This is a new area where the traditional applications of x-rays have been profoundly superseded. Examples are chosen from limited areas and are by no means exhaustive. The new emerging information will, no doubt, have great impact on materials science and engineering.

SUBTITLE

Introduction

Important new materials in industry are now being produced by far more sophisticated processing methods than previously utilized. These materials must be made with control of their buildup designed at the atomic level. Success with such materials requires the knowledge of new ways to arrange the atoms in order to achieve the desired results. But such knowledge, while important and necessary, is insufficient by itself. The structure of all materials when formed is non uniform locally often over regions of the order of a micrometer. Heterogeneity occurring as grain boundaries, phase interfaces, interacting dislocations, local compositional variations, regionally homogeneous strains (plastic deformation), and inhomogeneous strains, etc., often dominates the behavior of materials in sophisticated and/or demanding applications and severely weakens their performance. Therefore, as important as, or possibly even more important than, novel

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materials design will be information on, and control of, these local variations in actual materials produced by atomistic design—what Turnbull has termed "ultramolecular engineering" 1.

In fields other than electronics, the word "atomistic design" may sound extreme at the present stage of manufacturing technology. However, virtually all materials now used technologically are configurationally frozen from states that often were metastable, or even unstable before freezing. Moreover, some materials display non-equilibrium atomic level dispersions of impurities and topologically metastable structures. Sophisticated metastable synthesis has become quite common in industry due to newly developed techniques such as laser pulsing, ion implantation and rapid-cooling. Although one cannot, at present, design and control the arrangement of metastable states at will, current types of metastable synthesis certainly are the first steps toward atomistic design for industrial materials. Information on morphological grain and phase boundaries, strains between and within grains, and interacting dislocations must be obtained for materials performance as designed.

Heterogeneity can occur at very different levels when applied to different materials. In some instances it is the presence (concentration) of impurities that controls the quality of the product and its performance. For other materials, it is the spatial distribution of a particular phase or constituent that controls the properties of interest. For most materials, it is the presence of imperfections such as grain boundaries, subgrain boundaries, precursor cleavages, interacting dislocations, growth striations, and dendritic growth features. In any case, techniques that can establish an image of microstructure by some contrast mechanisms are essential 2.

### Synchrotron Radiation

In the past several years, an increasing number of synchrotron radiation beam lines have become available as new x-ray sources at storage rings and synchrotrons around the world. These new x-ray sources are quite ideal to obtain such images of the microstructure, once appropriate x-ray optical arrangements have been made.

Synchrotron radiation is typically produced by orbiting electrons or positrons in a storage ring. Charged particles, when constrained to move in a curved path, experience a centripetal acceleration and thus emit electromagnetic radiation over a wide solid angle. As the velocity of electrons approaches the speed of light, the pattern of radiation (Larmor pattern) is distorted by relativistic effects and exhibits a sharp directionality with extremely high flux. Normally the speed of charged particles is maintained constant in the storage ring (thus a centripetal acceleration is achieved) after being first accelerated to nearly the speed of light. The radiation is then confined to within an extremely thin and flat horizontal plane which contains the particle orbit in the storage ring. The vertical divergence  $1/\gamma$ , of the radiation (orthogonal to the storage ring plane) is given by the relationship,  $1/\gamma = 105/E(\text{GeV})$ , in arc seconds, where E is the energy of charged particles in unit of gigaelectronvolt.

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The energy of the continuous radiation spectrum moves into the hard x-ray energy region as the electron (or positron) energy increases into the gigaelectronvolt range with applications in condensed matter physics, crystallography, materials science, and dental and biomedical science. The unique features of synchrotron radiation are a continuous spectrum, high flux, brightness, intensity, and extreme collimation ( $1/\gamma$ ) in the vertical direction. This wide spectrum permits wave length tunability as described later. In addition, synchrotron radiation has a pulsed time structure (pulses of less than a nanosecond width with a microsecond interval) and a high degree of polarization in the plane of the storage ring. At most synchrotron facilities operated at the gigaelectronvolt range, the spectrum ranging from 3 keV (wavelength of about 4Å) to 60 keV (0.2Å) can readily be made available. The flux is of the order of  $10^{14}$  photons  $s^{-1}$  mrad $^{-1}$  mA $^{-1}$  in a 1% energy bandpass. While the low degree of vertical divergence of useful beams is given by the electron energy, the divergence in the orbital plane is determined by apertures in the beam transport and is typically several to tens of milliradians, which can be made narrower by additional slit systems.

A factor extremely important for imaging is the source size of synchrotron radiation in the storage ring. A small size, bright source permits the establishment of a highly parallel beam for high resolution imaging. Additional x-ray optical systems can also reduce the apparent source size of the radiation beam. The apparent source size is the actual source size observed (through any additional optical system) from single points on the sample. It is defined by an angle subtended to accept the radiation at these single points, and determines spatial resolution locally. Resolution is an important factor for imaging, and is related to the apparent source size, not to the divergence of the beam emitted from the source 3.

#### X-Ray Optics for Imaging

One of the main advantages to using synchrotron radiation is the wide continuous spectrum of x-rays, unmarred by characteristic x-ray lines, which can be used as a white radiation source. It also provides wavelength (or energy) tunability, that is, a choice of monochromatic radiation with various x-ray energies. Tunability can be established by inserting an x-ray optical system for monochromatization in the beam line. Heterogeneity and microstructure within materials can be imaged on film or video through a 2D image detector by a radiation beam prepared with or without the monochromator system, by use of various contrast mechanisms. For instance, the differences in absorption due to species of chemical elements and local density changes can be used to create image contrast even with a white radiation beam. This is radiography. In many instances, crystalline materials form a set of diffraction images with a white beam as well as monochromatic beams. Within each diffraction image, details of microstructure are revealed to exhibit grains, grain boundaries, phase interfaces, dislocations, and local strains. This process is called diffraction imaging or x-ray topography 2.

An important aspect in imaging is the capability to observe changes in images in real time: time-resolved observation. This is not only necessary for rapid inspection or survey of the quality of materials by scanning, but indispensable for the in situ observation of dynamic transient phenomena

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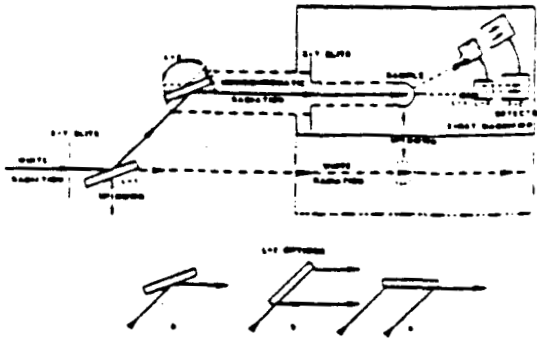


Figure 1. NBS Materials Science synchrotron beam line optics: Arrangement of flat crystal diffraction elements used for imaging. L1: monochromator first crystal, L2: monochromator second crystal (options b and c for imaging), L3: first crystal of x-ray magnifier, and L4: second crystal of x-ray magnifier (See, reference 5). Notice that the position of the beam exiting L2 is fixed, regardless of which energy is tuned.

REAL TIME  
TOPOGRAPHY / MICORADIOGRAPHY

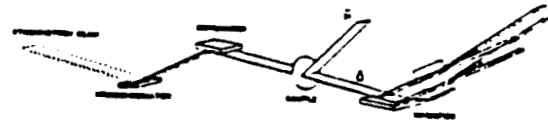


Figure 2. Image magnification: Schematic beam path for two dimensional magnification. This example shows the image formation for the 0(forward)-diffracted beam in transmission under the Bragg condition.

that take place within materials under environmental conditions. Although the current spatial resolution in real time imaging is tens of micrometers, efforts are underway to develop high resolution two dimensional detectors and to apply x-ray diffraction optics innovatively with submicrometer resolution in real time.

Figure 1 shows the x-ray optical arrangement for diffraction imaging, that has been used at the National Bureau of Standards materials science synchrotron beam line X-23A at National Synchrotron Light Source at Brookhaven National Laboratory 4,5. This arrangement provides for the versatile in situ real time application of synchrotron radiation to problems in many different areas of science and engineering. When white (polychromatic) radiation is desired as an incident beam, the first monochromator crystal, L1, is dropped by a computer command; and white radiation from the storage ring is delivered directly to a sample on a diffractometer, which is lowered by command to an appropriate position to accept the beam. The width and height of the beam can be adjusted by an x-y slit system.

In the monochromatic beam mode, the monochromator crystal, L1, is raised to intercept the white radiation beam from the storage ring. In response to a "tune" command that specifies the energy of x-ray photons, this crystal and another crystal, L2, move and rotate to the appropriate positions to give a monochromatic beam of the specified energy by flat crystal diffraction. The exit position of the beam remains at a fixed elevation, independent of photon energy. During the fulfillment of the tune command, the rocking curve of L2 crystal is automatically displayed on a

monitor to demonstrate the quality of the monochromatic beam. While observing the beam on the monitor, one can insert slits to control the beam size. After the slits are inserted, the tune command is repeated for this energy to optimize the x-ray optical trajectory for this beam in relation to the source. This process is important in diffraction imaging to ensure maximum brightness from the point within the source that provides the beam trajectory for a given optical arrangement.

It is important also to enable the selection of monochromatic beams of different dimensions and flux density. Monochromator crystal, L2, can be adjusted among three positions (See Figure 1); a) one for symmetrical diffraction (normally suitable for EXAFS), b) a second for asymmetric diffraction in the magnification mode (providing a large size, extremely parallel beam, suitable for diffraction imaging—topography), and c) a third for asymmetric diffraction in the demagnification mode (providing a concentrated, slightly focused beam, suitable for the use of an x-ray magnifier as described later, and/or for real time observation with enhanced flux). The choice of any of these three modes can be made any time by command while watching images, within the ranges of the geometry permitted by the L2 crystal.

A series of commands sets a sample in the desired position and orientation, and an imaging detector or analyzing x-ray optical system in a suitable position. The sample stage contains space sufficient to accommodate environmental chambers, such as a cryostat, furnace, tensile stage, or magnets. Radiographic (transmission) images and diffraction images in transmission or reflection are observed on the video monitor.

An optional x-ray image magnifier produces an undistorted image, magnified up to 150 fold before detection by either film or an image detector 6,7. Even with the current limitation in the spatial resolution of two dimensional image detectors, one can reach the submicron range of resolution in real time. As shown in Figure 1, magnifier crystals L3 and L4 are placed orthogonally to produce two dimensional magnification by two successive stages of asymmetric Bragg diffraction. Figure 2 illustrates the formation of a two dimensionally magnified image of a 0-diffracted (forward diffraction) beam in transmission. This x-ray magnifier can be used as a zoom lens, since a given pair of two asymmetrically cut crystals, such as L3 and L4, provides continuously changing magnification factors as the energy of the incident beam is changed by the monochromator system.

The use of flat crystal diffraction in the analyzer stage provides additional important features for diffraction as well as microradiographic imaging. For example, crystal L3 alone can act as an ideal slit with an aperture less than an arc second through asymmetric diffraction. This defines precisely the momentum transfer of x-ray photons in diffraction and scattering; in other words, the scattering angle can be set with an accuracy much less than one arc second. With an incident beam that is prepared to be extremely parallel by the monochromator system described above, the analyzer crystal is thus capable of providing for equi-d-spacing mapping, angle-resolved imperfection imaging, and small angle x-ray scattering (SAX) imaging 8-12. These analytic features arm diffraction imaging (topography) with a truly quantitative capability in which the spatial information in images is preserved.

The x-ray optical system described above is versatile, in that this system can be used in the manner of a conventional diffractometer ( $\theta$ - $2\theta$  scan) as well as in the manner to scan the  $q$ (momentum)-space in diffuse scattering analysis. In addition, the high flux, extreme brightness, and high degree of parallelism of the synchrotron x-ray beam that is prepared by the x-ray optical system described provide a unique opportunity to tackle challenging materials science problems by techniques hitherto considered impractical. In particular, microstructural effects can be observed and measured in situ on a real time basis, even under simulated environmental conditions. Some examples of these applications to microstructural imaging will be described in the following sections for white radiation as well as for monochromatic radiation.

#### Examples of White Radiation Imaging

When a white beam of x-rays is incident on a sample, the direct beam contains the microradiographic image of the crystal sample, while diffracted beams form a Laue pattern. Each spot of the Laue pattern is of roughly the same size as the beam on the sample. Within each spot can be observed a fine structure related to the microstructure in the sample. These microradio-graphic and diffracted images are recorded on an imaging detector serving as a video monitor and, if the images are of particular interest, they can be recorded on high resolution film. The experimental simplicity of Laue diffraction imaging lies in the fact that grains of arbitrary orientation are imaged as the sample selects an appropriate wavelength that satisfies the Bragg condition locally. Studies with white radiation have been performed on the recrystallization, and subsequent coarsening of grains of silicon iron, aluminum and several alloys 13-20.

The example shown in Figure 3 is a result of such a study in the real time recording of the recrystallization process of a deformed aluminum sample. This figure shows approximately half of the transmission Laue pattern photographed from the video monitor at various times and temperatures during heating. Fifteen minutes after rapid heating to  $300^{\circ}\text{C}$  and subsequent slow heating to  $325^{\circ}\text{C}$ , the sample yields diffuse streaked Laue spots indicating a deformed state and no signs of freshly formed grains. In the sequence following this photograph, the nucleation and growth of strain-free grains is quite evident. The process of primary recrystallization is essentially finished after approximately 30 minutes, when the coarsening process takes place thereafter. An analysis of changes in area of individual grain images as a function of time was made by computer analysis of the video signal. The result was compared with a current theory of Ostwald ripening 16.

Another example of the coarsening of alloy liquid-solid mixtures of aluminum alloys with 5 weight percent tin demonstrates the selected area diffraction technique coupled with microradiography, as shown in Figure 4. In this in situ observation, the radiograph shown in Figure 4a was observed on the video monitor. A roughly  $100\mu\text{m}$  aperture was then positioned sequentially over the numbered grains, and transmission Laue patterns were recorded on film and video tape as shown in Figures 4 b, c and d. This study provides statistical microstructural and crystallographic orientation data that show the coarsening to be controlled by diffusion in the liquid phase even though liquid exists only at the grain edges 17. This technique

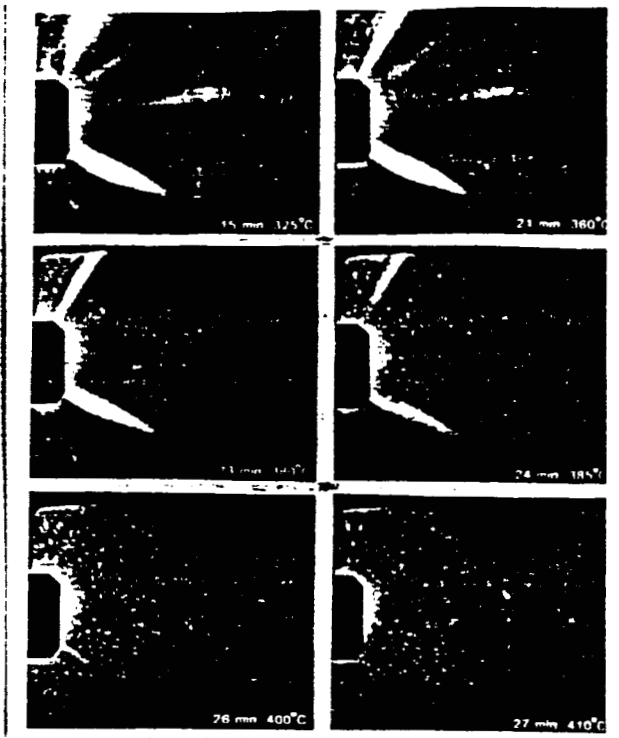


Figure 3. Recrystallization of a deformed aluminum disk: Photographs of the video monitor showing half of the Laue pattern from recrystallizing Al for the times and temperatures indicated. (Reference 16)

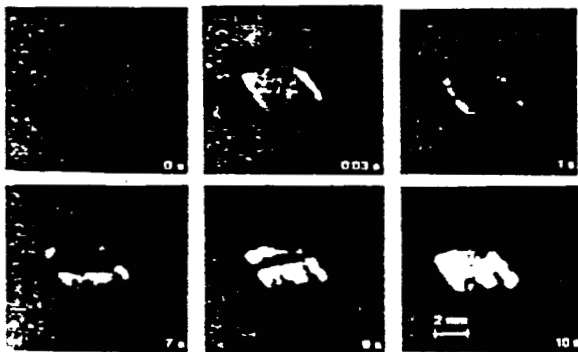


Figure 5. Solidification of Sn observed in a white radiation Laue spot: A video sequence of topographs during the liquid-solid transition of tin undercooled at 230° C. After one second, the sample recalesces, and the entire sample solidifies to become a twin as the liquid-solid interfaces move in from two directions. (Reference 10)

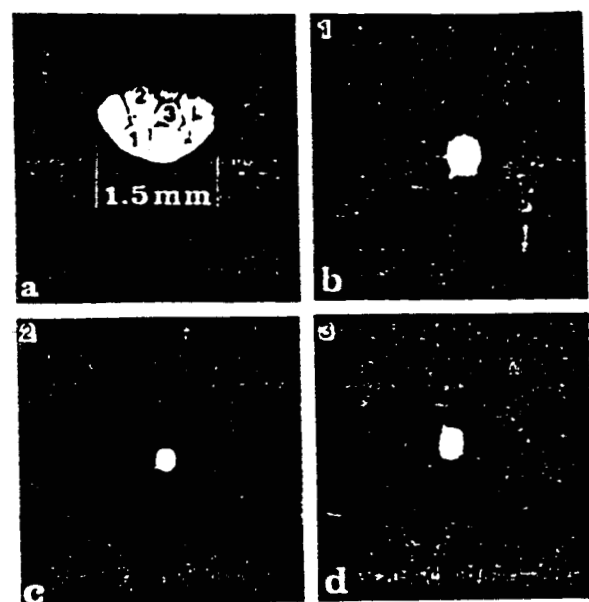


Figure 4. Selected area diffraction obtained from specified grains viewed on the video monitor: (a) White radiation microradiograph with various grains numbered, (b, c, d) Transmission Laue diffraction patterns of the numbered grains. (Reference 17)



Figure 6. Comparison of white radiation images with a monochromatic diffraction image: a) Monochromatic surface (004) reflection, b) (062) Laue spot image, c) (220) Laue image, d) (040) Laue image.



is useful in the understanding of various microstructural phenomenon such as liquid film migration at grain boundaries 21.

In the real time observation of Laue spots during the freezing and melting of a tin crystal (Figure 5), the motion and shape of the liquid-solid interface and the surface tension at the interface can be measured, along with the observation of recalescence 10. The Laue technique can be used also for non-single crystalline materials, such as rapidly cooled alloys consisting of particles 100  $\mu\text{m}$  in diameter or less. This observation not only shows the degree of uniformity in the materials, but also gives information on nucleation in individual particles for the improvement of processing 22. Similarly, the application of this technique to superalloy turbine blades has assisted prediction of the life-time in fatigue 22.

As described above, white radiation diffraction imaging (topography) is simple and useful for the simultaneous observation of Laue spots from materials containing different grains and variously oriented diffracting planes. The determination of dislocation Burgers vectors in the study of active slip systems demonstrates the unique role of this imaging technique. The ease in implementation of this technique is an important factor in its popularity. However, the analysis of these images is very complex because of the range of wavelengths even within a single Laue spot. This range reduces sensitivity to crystalline strain and spatial resolution from those attained with monochromatic imaging. Since image contrast, that is the intensity distribution in the image, is an important factor in analysis, white radiation images are not suitable for advanced quantitative analysis. Because of these sources of image degradation white radiation topography alone does not support the detailed determination of microstructure and defects in materials.

#### Examples of Monochromatic Radiation Imaging

Underlying x-ray diffraction imaging is the x-ray extinction effect 23, 24. Fine structures within diffraction images are the result of the reduction of primary and secondary extinction due to crystal imperfection (deviation from the ideal lattice periodicity) 2, 10. As changes in primary and secondary extinction cause intensity variations and diffraction line broadening in crystallography, so these same phenomena manifest themselves by diffraction in different local regions, resulting in contrast (intensity distribution) in the diffracted images of x-ray topography. For maximum advantage of the known x-ray optical mechanisms in diffraction for data analysis, a high degree of monochromaticity and parallelism is desirable in the incident beam. As described in a previous section, a monochromatic beam can be prepared with relative ease for low divergence (less than an arc second) and a beam cross section (5 cm X 5 cm) for a choice of photon energies.

The following example, taken from a 24 atomic percent iron-aluminum alloy, illustrates the superiority in strain sensitivity of monochromatic diffraction imaging over white radiation imaging. After a Laue topograph is obtained and indexed, enlarged views of several Laue spots are inspected for the dependence on Miller indices, as shown in Figure 6b, c and d. These images clearly show grain boundaries and "dumbbell" images typical of spherical inclusions. In addition, the existence of other imperfections is



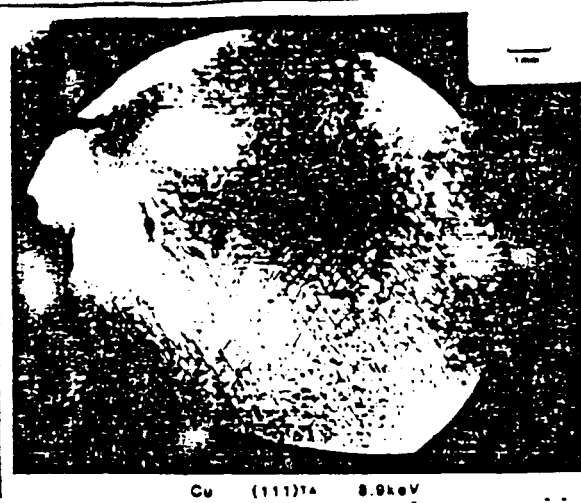


Figure 7. Monochromatic beam quality: This asymmetric (111) transmission image from a single crystal of copper 0.8 mm thick shows individual dislocations. (Reference 25)

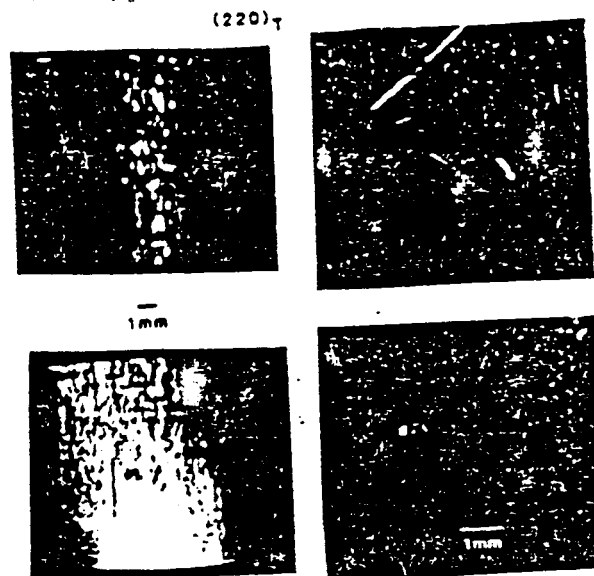


Figure 8. Comparison of video images with the images on high resolution film.

noticeable, and prompts a more detailed study of this sample by monochromatic radiation topography, as shown in Figure 6a. There, subgrain structures and magnetic domains are clearly revealed. For improvement in the current quality of single crystal materials, of such electro optical materials as III-V, II-VI and compound materials, and for research aimed at a fundamental understanding of the nature of interfaces, monochromatic radiation topography is essential. The superb quality of monochromatic radiation diffraction images is shown in a transmission topograph taken from a copper single crystal 0.8 mm thick (Figure 7) 25.

The crystal quality of advanced materials can be assessed effectively in real time by monochromatic radiation diffraction imaging. Figure 8 shows a comparison between images on the video monitor and those on high resolution film. Major defects are as recognizable on the monitor views as on film. Scanning the sample under view in real time is a remarkable capability provided by synchrotron radiation. In particular, a diffraction image seen at a given glancing angle is by no means the same as images seen at different glancing angles, as shown in Figure 9, even if these angles are in the immediate vicinity of the Bragg angle. The real time rocking as well as scanning capabilities are indispensable for microstructural characterization. For example, the series of images in Figure 9 give depth profiling of imperfections 26.

The crystal growth conditions of materials determine the quality of the crystals grown, and the effects of various device fabrication processes can be investigated. As seen in many examples of Si, 27, GaAs, 28-30, CdTe, 30, lithium niobates, 30, bismuth silicates, 26, mercuric iodide, 30, monochromatic diffraction imaging has contributed important information to their crystal growth process. Some of the imperfections in these materials have not been observed previously by traditional methods. For example, a

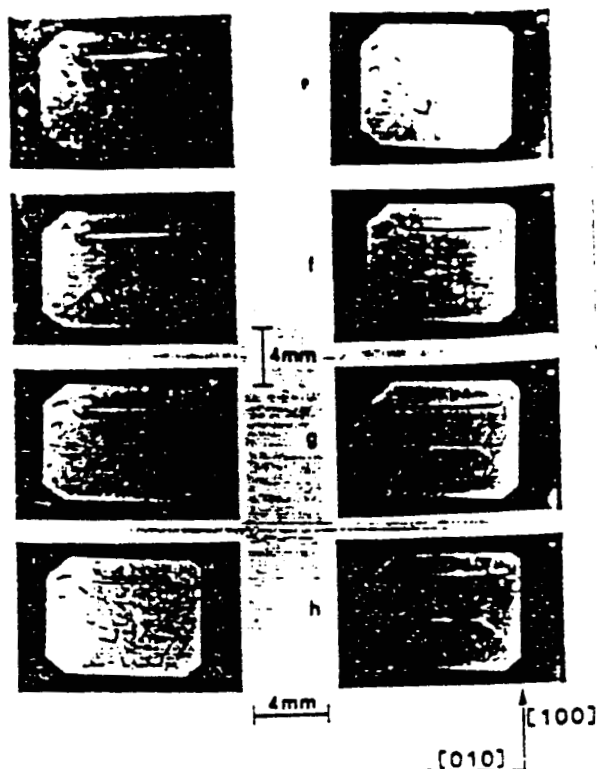


Figure 9. Rocking curve equivalent of video images from a bismuth silicate crystal: These Bragg geometry topographs are excerpts from the video tape of the 006 diffraction, taken while the crystal was rocked over the entire angular region (acceptance angle—rocking curve) of diffraction. (Reference 26)

Figure 11. Transmission images of a bismuth silicate crystal with 13.4 KeV monochromatic radiation tuned to slightly below the L-III absorption edge of Bi: (030) diffraction is used, the O image is diffracted parallel to the incident beam direction, and the H image is diffracted in the Bragg direction. H indicates the scattering vector direction. (Reference 26)

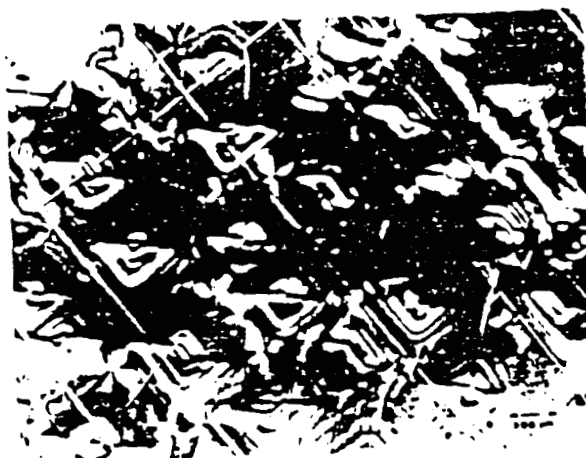
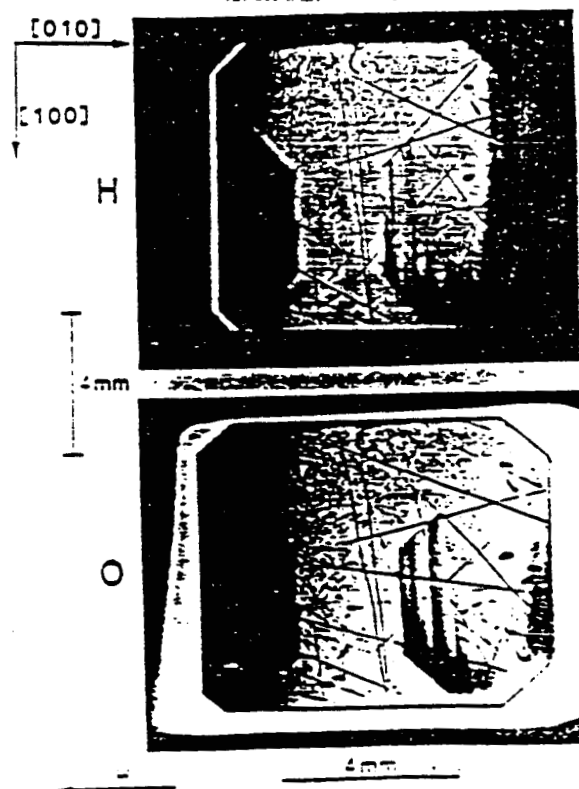


Figure 10. Facet-growth region of In-doped GaAs: Enlarged portion of a transmission topograph (040 diffraction) of a 0.8 mm thick indium doped gallium arsenide crystal with 10 keV monochromatic radiation.



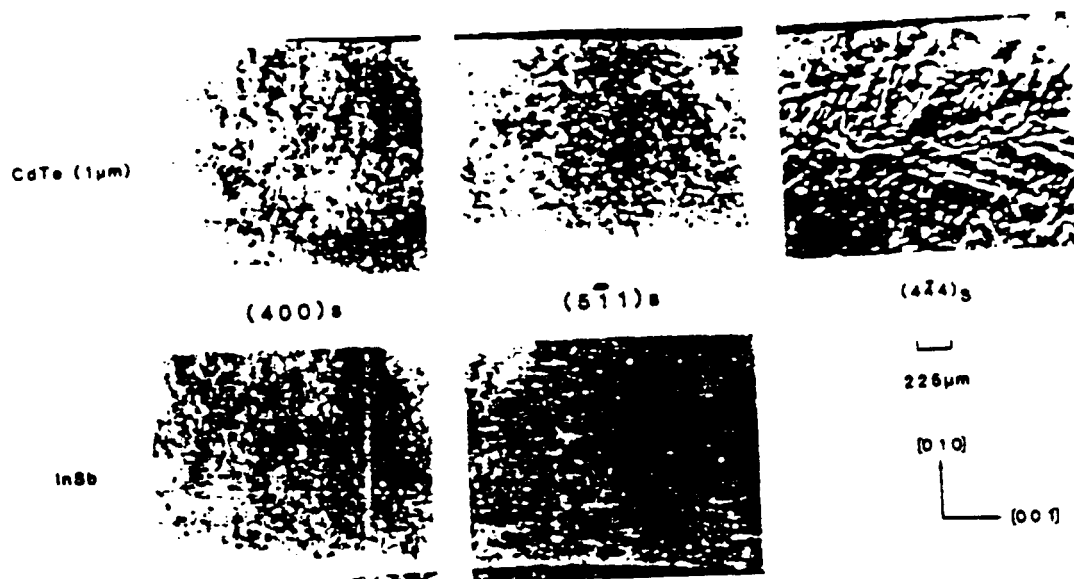
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transmission topograph of a faceted growth region of an indium doped gallium arsenide is shown in Figure 10. In the study of a bismuth silicon oxide boule, the reconstruction of the crystal growth from various sets of diffraction images was completed successfully, aided by the tunability of synchrotron radiation 26. By tuning the photon energy to slightly below the Bi L-III absorption edge, transmission topographs were obtained from sample slices 0.7 mm thick, as shown in Figure 11. The growth striations were identified as successive epitaxial deposition layers of atoms making the  $45^\circ$  angle to the pulling direction of the boule, and the development of five different segments whose mutual misorientations were within one arc second was elucidated.

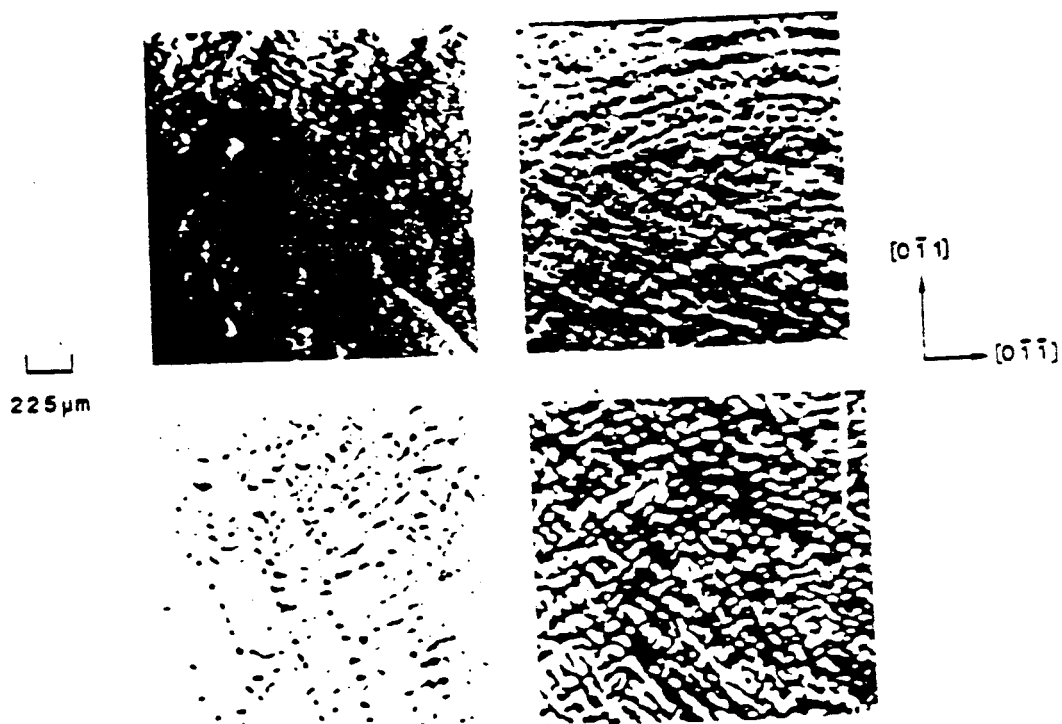
For multilayered materials, the high flux of monochromatic radiation plays a unique role. This radiation can impinge on the sample at an extreme glancing angle, and permit the grazing-angle diffraction or total reflection with respect to substrate crystals for the study of the surface or interfacial regions of the film on the substrate. A simplest example is shown in Figure 12, where asymmetrical diffraction, normally unavailable with the ordinary x-rays, can be excited to detect the interfacial strains between a CdTe film and an In-Sb substrate. The nonuniformity of this film is easily noticeable in asymmetric diffraction.

For quantitative measurements of strain fields around imperfections and near interfaces, a series of topographs is required both as a function of incident glancing angle and of observation (detecting crystal) angle 8, 31, 32, 33. This technique is called "angle-resolved imperfection scattering" or "in and out states analysis", where the scattering from imperfections can be separated from the dynamical scattering from the matrix material. These images are stored on video tape for subsequent quantitative analysis. Experiments were performed with a copper crystal with Knoop indentations for analysis of strains associated with indentation impressions 11, 34, 35 and with an indium doped gallium arsenide for the formation of growth facet striations associated with variations in indium concentrations. Without synchrotron radiation, it is impossible to obtain such a series of two-dimensional data within a feasible time frame. This approach, in a general line-broadening context, opens up a new avenue of characterizing imperfections locally and quantitatively in terms of a "structure factor" containing local atomic displacements.

The current status of diffraction imaging indeed makes possible real-time observations of local phenomena (microstructural changes) within materials, during phase transitions, tensile testing and even crystal growth. Michot and coworkers studied the development of dislocations associated with a crack, as part of kinetic study of fracture 36, 37. This experiment was carried out in quasi-real-time, where images were taken on a high resolution plate in a sequence every 300 seconds or longer, as an initial crack from a notch developed in a silicon wafer at  $750^\circ\text{C}$  under a load. The results showed the development of dislocations and both primary and secondary slip systems as a function of time. Another typical example is the real-time observation of melting and growth of silicon crystals by Chikawa and Sato, 38. Dislocations are seen to be extremely mobile in the vicinity of liquid-solid interface. There have been an increasing number of studies of the kinetics of crystal growth and of fracture mechanics, 17-20, 39, 40, although most of them were limited by white radiation imaging.



a  
(444)<sub>s</sub>



b

Figure 12. CdTe film on InSb: Interfacial strain between film and substrate is detected only in the highly asymmetric 444 surface diffraction. Local variations of microstructure is evident.

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Monochromatic diffraction imaging is certainly an important new area in materials science and engineering, one enabling real time observations and measurements in a more comprehensive way than previously practiced. A principal need now is improvement in the spatial resolution of real time observations. To circumvent the present limit of spatial resolution (about 10  $\mu\text{m}$ ) of imaging detectors, the x-ray magnification technique has been developed, as described in a previous section 6, 7. Currently, this technique is effectively being applied to microradiography for dental research, 5, 41, 42, where conventional contact microradiography has been of limited use and impossible for real time in situ observations. When applied to diffraction imaging, x-ray image magnification (up to 200X) before detection makes it possible to observe dynamic events within materials under simulated environmental conditions with submicron spatial resolution and high sensitivity. Three-dimensional tomosynthetic image reconstruction is no longer a dream not only for microradiography but for diffraction imaging. Biomedical applications, such as angiography, are natural extensions.

The use of diffraction imaging provides new knowledge on microstructural details and kinetic behavior quantitatively as well as visually on a real time basis. The areas discussed above represent only examples of the potential of diffraction imaging. Similar applications to other problems in materials science can be made with little modification. Clearly the use of synchrotron radiation will lead to an understanding of many problems in materials science at the atomic level.

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